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Graphene oxide modified polypropylene fiber filter membrane for enhanced treatment efficiency of water-based paint wastewater

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ABSTRACT

In this study, graphene oxide (GO) was used to modify the polypropylene fiber filter membrane. The GO modified polypropylene fiber filter membrane (GO-PP fiber filter membrane) was obtained using vacuum filtration. The performance of the GO-PP fiber filter membrane was tested to filter the practical water-based paint wastewater. GO-PP fiber filter membrane has better treatment effect and stronger anti-pollution performance than commercial PP fiber filter membrane. Chemical oxygen demand (COD), membrane flux and suspended solids (SS) were used as indicators to characterize the performance of the GO-PP fiber filter membrane. The combination process of flocculation and PP fiber filter membrane for wastewater treatment was studied. The optimal COD and SS removal rates were obtained using flocculation + GO-PP fiber filter membrane unit as 84.8% and 84.6%, respectively. These results suggest that the GO-PP fiber filter membrane could be a potential material for water-based paint wastewater treatment.

Keywords: Water-based paint wastewater; Graphene oxide; Polypropylene fiber filter membrane; Flux; Flocculation

1. Introduction

Water-based paint was a kind of coating material, which has an important position in many traditional industries, including automobiles, ships, petroleum equipment and chemical equipment [1,2]. The discharge of untreated water-based paint can cause environmental pollution and other problems [3,4]. With the development of the manufacturing industry, the treatment of water-based paint waste liquid has received extensive attention. Therefore, it was of great significance to find an efficient and economical treatment method.

Many methods have been developed to treat water-based paint containing wastewater, including membrane filtration [5–7], adsorption [4,8], biodegradation [9], flocculation/coagulation [10,11] and advanced oxidation [12–14]. Filter technology was an important method in a series of water treatment processes because of its simplicity and high efficiency [15–18]. However, the efficiency of the filter process was often limited by the filter material, and the existing commercial membrane filter have defects such as low flux, poor anti-fouling ability, and poor chemical resistance. Polypropylene fiber filter membrane is a common commercial filter membrane, which have been widely employed in the fields of gas adsorption [19,20], heavy metal [21], organic [22–24] and inorganic salt [25,26] treatment. At present, in order to improve the filter process, some studies have taken the development and application of composite filter materials as a breakthrough point. Graphene oxide (GO) modified membrane filter has attracted more attention because of their superior lamellar structure, efficient separation performance and flexible preparation methods [27–30].

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Due to the complex and stubborn nature of industrial wastewater, the single treatment method cannot effectively treat the pollutants in wastewater, which highlights the need and development of mixed processes [31]. Studies have indicated that the synergy between flocculation/coagulation and membrane filter was feasible for the treatment of industrial wastewater, due to the advantages of high removal efficiency for high-concentration pollutants, low sludge production, increased organic matter removal rate, and less membrane pollution [32–38]. Therefore, the combination process of GO modified PP fiber filter membrane and flocculation can increase the purification rate of water bodies. Jin et al. [39] prepared a polymer-based graphene oxide composites membrane with excellent hydrophilicity and antifouling performance, which shown 100% rejection of protein and high flux for the protein solution.

In this study, GO was prepared by the Hummers method. The obtained GO was used to modify commercial PP fiber filter membrane using vacuum filtration. The treatment efficiency of actual wastewater using prepared GO modified PP fiber filter membrane was tested. Chemical oxygen demand (COD) removal rate, membrane flux and suspended solids (SS) were used as the water quality index. Besides, combination technology of GO modified PP fiber filter membrane and flocculation for wastewater treatment was investigated.

2. Experimental

2.1. Materials and instruments

2.1.1. Materials

PP cotton filter was purchased on Super Water Purification Co., Ltd., Hangzhou, China, (membrane area: 0.6 m^2, diameter: 69 mm). High purity graphite powder purchased from Sinopharm Chemical Reagent Co., Ltd., (Shanghai, China). Other reagents include concentrated sulfuric acid, concentrated hydrochloric acid, potassium permanganate, sodium nitrate, mercury sulfate, hydrogen peroxide, polyaluminum chloride, silver sulfate and potassium dichromate. All reagents are provided by Sinopharm Chemical Reagent Co., Ltd., with pure grade.

2.1.2. Instruments

The morphology and surface elemental compositions of sample was recorded using emission scanning electron microscope (SEM, JSM-IT300), the samples were previously coated with a thin layer of gold to conduct electricity and the micrographs were generated by topographic contrast using an electron microscope under standard high vacuum conditions. Other instruments include digital display electric heating constant temperature water bath (HWS12), COD rapid tester (Suntech SN-200), ultrasonic cleaner (KQ-3200E), peristaltic pump (BT300-2J), turbidity meter (WGZ-200) and pH meter (PHS-3D).

2.2. Preparation of GO solution

The improved Hummers method was used to prepare graphene solutions, which was divided into three stages.

Firstly, 115 mL of concentrated sulfuric acid and 2.5 g of sodium nitrate were added to the beaker, and then the beaker was placed in an ice-water bath with constant stirring, and the temperature of the reaction was controlled below 5°C.

Secondly, a certain amount of graphite powder and potassium permanganate was added to this beaker and reacted at 60°C for 30 min. Then, 700 mL of deionized water and 25 mL of hydrogen peroxide were added to the beaker. When the color of the solution turns yellow, the solution was filtered and washed with dilute hydrochloric acid, and then dried at 60°C for 24 h to obtain graphene oxide powder.

Thirdly, to obtain GO solution, graphene oxide powder was diluted using pure water with a concentration of 1 g/L and then placed in an ultrasonic machine for 1 h.

2.3. Preparation of GO modified PP fiber filter membrane

The PP fiber filter membrane was immersed in 1 g/L graphene oxide solution for 24 h. The PP fiber filter membrane was flipped every 6 h to ensure the even dispersion of GO on PP fiber filter membrane. Then, vacuum filtration was used to prepare the PP fiber filter membrane. The prepared GO modified fiber (GO-PP fiber filter membrane) membrane was dried in dryer at 60°C for 24 h. Fig. 1 shows the photographs of GO-PP fiber filter membrane and PP fiber filter membrane.

2.4. Application of water treatment device

The practical wastewater used in this study was collected from Shiyan Dongfeng Automobile Factory (Shiyan, China) and the water parameter was illustrated in Table 1. Self-made filter unit was employed for water treatment (Schematic diagram shown in Fig. 2). In the process of operation, peristaltic pump was employed to adjust the inlet water flow. The performances of PP fiber filter membrane and GO-modified PP fiber filter membrane were both investigated. The filter

Fig. 1. Photographs of PP fiber filter membrane (left) and GO-PP fiber filter membrane (right).

Table 1
Characteristics of water-based paint wastewater

COD (mg/L)	1,103
SS (mg/L)	212
pH	8.2
Turbidity (NTU)	921

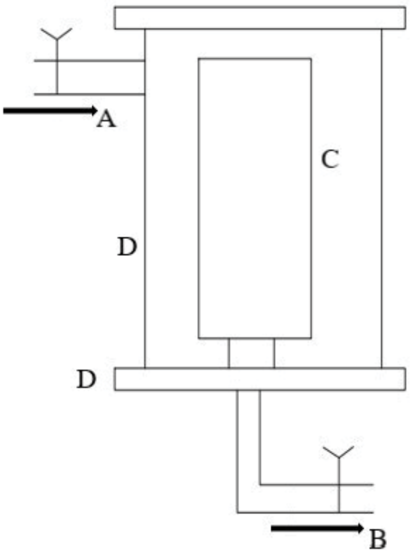

Fig. 2. Schematic diagram of filter unit (A: inlet; B: outlet; C: filter and D: Plexiglass board).

unit adopts a continuous operation mode and effluent was taken at the interval of 2 h. After running for 24 h, equipment was backwashed using pure water.

2.5. Analysis method for COD, turbidity, pH, membrane flux and SS

SS was analyzed according to national standard-determination of suspended matter in water gravimetric method (GB 11901-1989). COD was tested according to national standard-determination of chemical oxygen demand of water quality in potassium dichromate method (HJ 828-2017). Turbidity was tested according to national standard-determination of turbidity in waterbody (GB 13200-91). pH was tested according to national standard-determination of pH in waterbody (GB 6920-86). COD removal rate and membrane flux are calculated according to the following equations:

$$R = \left(1 - \frac{C_0}{C_i}\right) \times 100\% \tag{1}$$

$$J = \frac{V}{A_m t} \tag{2}$$

where R is COD removal rate (%); C_0 is COD concentration of inlet (mg/L); C_i is COD concentration of outlet (mg/L); J is membrane flux (L/(m^2 h)); V is volume of inlet (L); A_m is membrane flux (m^2) and T is operation (h).

3. Results and discussion

3.1. Pretreatment of water-based paint wastewater

In the pretreatment process of water-based paint wastewater, polyaluminum chloride (PAC) was used as flocculent due to the merit of high efficiency and low cost. The optimal dosage of PAC was determined according to the batch experiment (shown in Fig. 3). Fig. 4 shows the photograph of pretreated and untreated wastewater. The dosage of PAC was selected as 3 g/L due to the relatively high removal rate of COD, SS and turbidity. Excessive addition of flocculant would lead to generation of more flocculates and increase the treatment cost. With 3 g/L dosage of PAC, the removal rate of COD and SS using PAC was 53.6% and 52.2%, respectively.

3.2. Removal rate of COD

In the experiment section for COD removal and membrane flux, the filter membrane equipment was continuously operated for three times (0–24 h, 24–48 h, 48–72 h). After operation for every 24 h, filter membrane equipment was backwashed using pure water.

Fig. 5a shows the removal rate of COD by the PP fiber filter membrane. In the initial stage of operation, the removal rate of COD (operation for 2 h) was 54%, and the removal rate was 41% after operation for 24 h; the removal rate was 38.7% and 40% after operation for 48 h and 72 h, respectively.

Fig. 5b shows the removal rate of COD by GO modified PP fiber filter membrane. In the initial stage of operation, the removal rate of COD (operation for 2 h) was 58%, and the removal rate was 53% after operation for 24 h; the removal rate was 55% and 52% after operation for 48 h and 72 h, respectively.

Fig. 5c shows the removal rate of COD by the combination process of flocculation and PP fiber filter membrane. In the initial stage of operation, the removal rate of COD (operation for 2 h) was 76.6%, and the removal rate was 72.6% after operation for 24 h; the removal rate was 75.6%, 76.6% after operation for 48 h and 72 h, respectively.

Fig. 5d shows the removal rate of COD by the combination process of flocculation and GO-PP fiber filter membrane. In the initial stage of operation, the removal rate of COD (operation for 2 h) was 84.6%, and the removal rate was 80.9% after operation for 24 h; the removal rate was both 80.6% after operation for 48 h and 72 h, respectively.

The GO-PP fiber filter membrane has a certain improvement in the removal effect of COD from water-based paint waste liquid compared to the PP fiber filter membrane. The addition of GO to the surface of the membrane allows other removal mechanisms to occur, such as size exclusion, electrostatic repulsion, and adsorption. These mechanisms optimize the selectivity of the membrane, enhancing pollutants removal rate, and reducing fouling [40]. Therefore, the GO film on PP fiber filter membrane has a certain removal effect on the organic solutes in the water-based paint wastewater. The removal rate of COD after backwashing was lower than the initial removal rate, indicating that backwashing cannot completely remove the contaminants on the membrane surface, but it can effectively improve the service life of PP fiber filter membrane. Flocculation has contributed 53.6% of COD removal rate, indicating the strategy of flocculation combined with PP fiber filter membrane was obviously better than that of membrane filtration alone. The flocculation combined with GO modified PP fiber filter membrane shows better performance for COD removal.

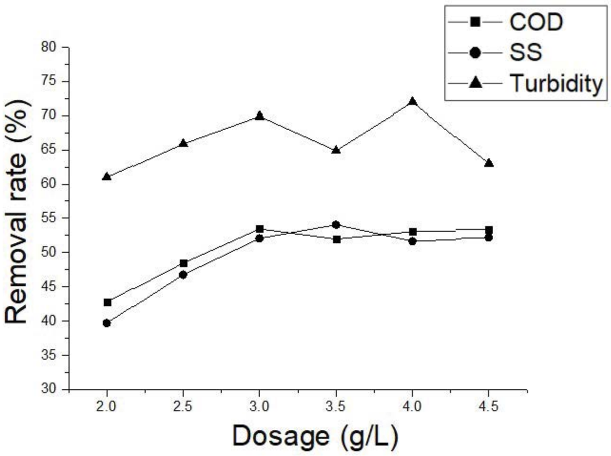

Fig. 3. Change of removal rate with varying dosage of PAC.

Fig. 4. Comparison of water-based paint waste before (left) and after (right) flocculation.

3.3. Membrane flux

Fig. 6a shows the membrane flux change during operation using PP fiber filter membrane. In the initial stage of operation, the membrane flux (operation for 2 h) was 160 L/

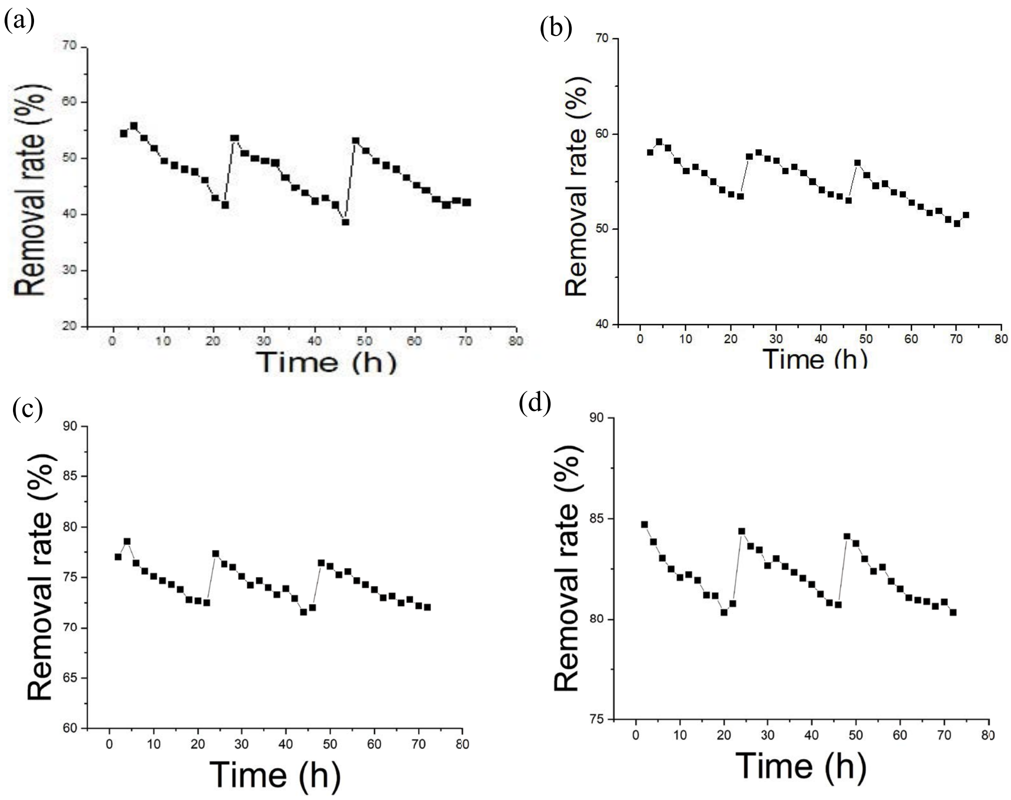

Fig. 5. COD removal rate under varying unit: (a) PP fiber filter membrane, (b) GO-modified PP fiber filter membrane, (c) flocculation + PP fiber filter membrane and (d) flocculation + GO-modified PP fiber filter membrane.

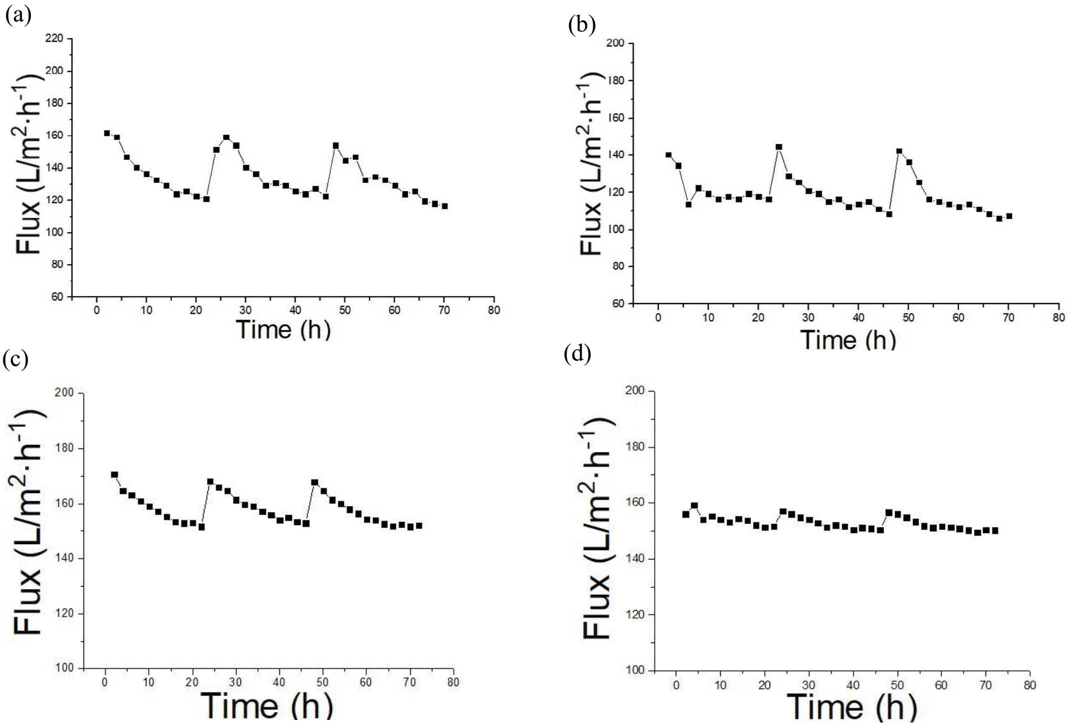

Fig. 6. Flux under varying unit: (a) PP fiber filter membrane, (b) GO-modified PP fiber filter membrane, (c) flocculation + PP fiber filter membrane and (d) flocculation + GO-modified PP fiber filter membrane.

(m^2 h), and the membrane flux was 120 L/(m^2 h) after operation for 24 h; the membrane flux was both 120 L/(m^2 h) after operation for 48 h and 72 h, respectively.

Fig. 6b shows the membrane flux change during operation using GO-PP fiber filter membrane. In the initial stage of operation, the membrane flux (operation for 2 h) was 140 L/(m^2 h), and the membrane flux was 115 L/(m^2 h) after operation for 24 h; the membrane flux was both 105 L/(m^2 h) after operation for 48 h and 72 h, respectively.

Fig. 6c shows membrane flux change during operation using the combination process of flocculation and PP fiber filter membrane. In the initial stage of operation, the membrane flux (operation for 2 h) was 170 L/(m^2 h), and the membrane flux was 151 L/(m^2 h) after operation for 24 h; the membrane flux was both 153 L/(m^2 h) after operation for 48 h and 72 h, respectively.

Fig. 6d shows membrane flux change during operation using the combination process of flocculation and GO-PP fiber filter membrane. In the initial stage of operation, the membrane flux (operation for 2 h) was 156 L/(m^2 h), and the membrane flux was 150 L/(m^2 h) after operation for 24 h; the membrane flux was 152 L/(m^2 h) and 150 L/(m^2 h) after operation for 48 h and 72 h, respectively.

The results indicate that the membrane flux to the GO-PP fiber filter membrane was reduced compared with that of PP fiber filter membrane, which was ascribed to the

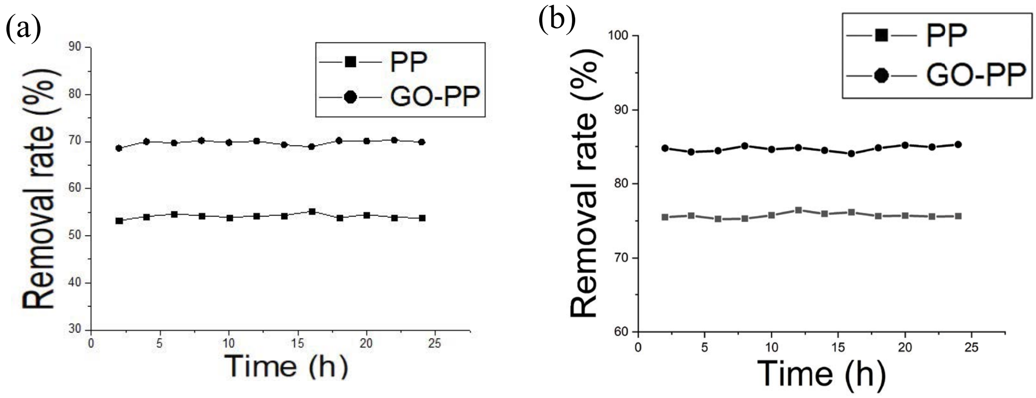

Fig. 7. SS removal rate under varying unit: (a) PP fiber filter membrane and GO-PP fiber filter membrane, (b) flocculation + PP fiber filter membrane and flocculation + GO-PP fiber filter membrane.

internal connections blockage between pores on the surface of PP fiber filter membrane and additional resistance caused by the GO layer [41,42]. Backwash could effectively restore the membrane flux. In the combination process of flocculation and PP fiber filter membrane, GO-PP fiber filter membrane was slightly lower than that of the PP fiber filter membrane while the GO-PP fiber filter membrane exhibited stronger stability of membrane flux, indicating the stronger anti-pollution performance of GO-PP fiber filter membrane.

3.4. SS removal analysis

In one operating cycle, the removal rate of SS by the four units was basically stable, and the effect of GO-PP fiber filter membrane was significantly better than that of PP fiber filter membrane.

Fig. 7 and Table 2 show the removal rate of SS under different units. In the initial stage of operation, the removal rate of SS (operation for 2 h) was 53.2%, 68.6%, 75.5% and 84.8% for PP fiber filter membrane, GO-PP fiber filter membrane, flocculation + PP fiber filter membrane and flocculation + GO-PP fiber filter membrane, respectively. After operation for 24 h, the removal rates of SS in the four units were still relatively stable. The result indicated the PP fiber filter membrane had a certain of removal efficiency for SS and the incorporation of GO could enhance the removal efficiency of SS. In addition, the backwash for membrane was unnecessary for SS removal in relative short operation time.

3.5. SEM-EDS analysis

Fig. 8 illustrates the SEM-EDS characterization of used PP fiber filter membrane and used GO-PP fiber filter membrane. It can be concluded that the surface of the GO-PP fiber filter membrane element was contaminated and the scale was covered by a layer of resin. The C and O elements are mainly derived from the graphene oxide film on the surface of the filter element, polypropylene in the film material, and water-based paint molecules. The GO-PP fiber filter membrane contains Cu, S and other elements, which are mainly derived from water-based paint liquid and other additives; Al, Fe, Si, Ca and other elements are derived from the mineral pigments in the water-based paint wastewater.

4. Conclusion

GO-PP fiber filter membrane was prepared successfully using vacuum filtration. The incorporation of GO could enhance the COD and SS removal rate using PP fiber filter membrane. In addition, the combination process of GO modified PP fiber filter membrane and flocculation can increase the purification rate of water bodies. The COD

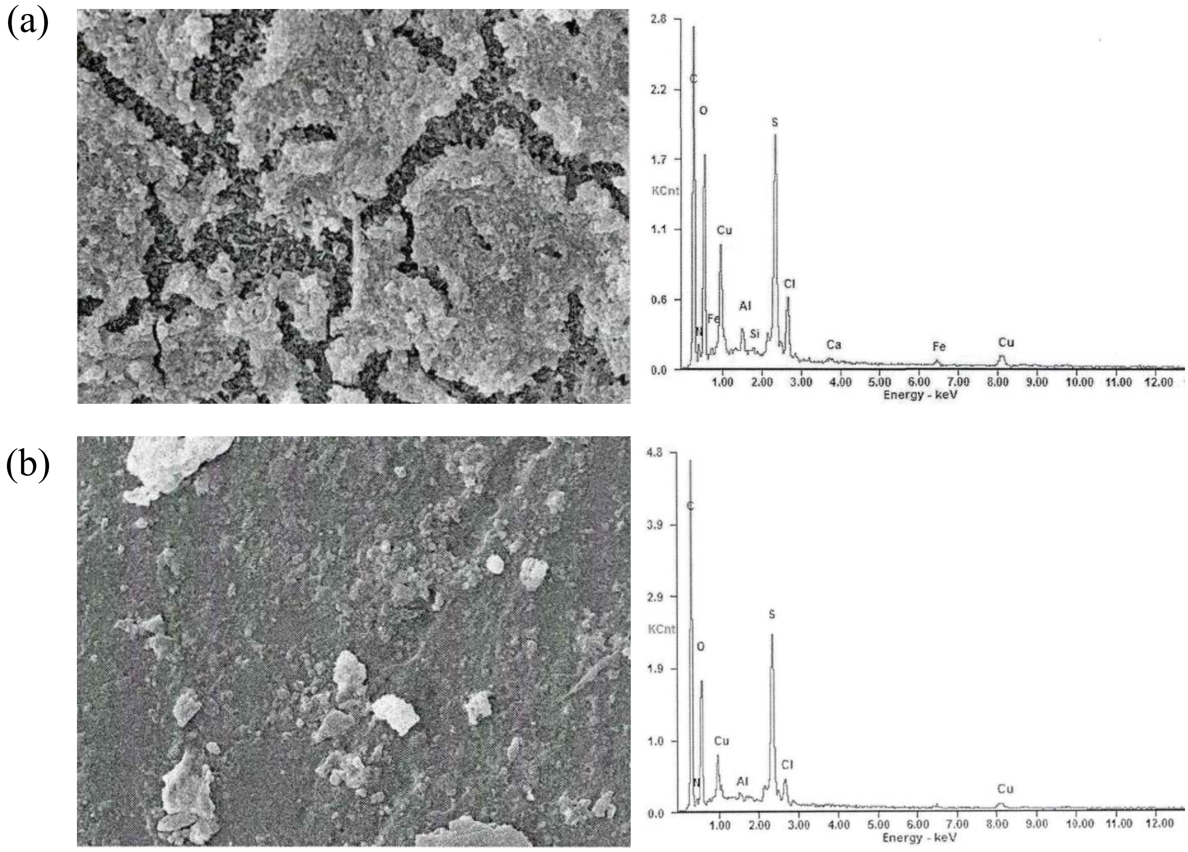

Fig. 8. SEM-EDS characterization of (a) GO-modified PP fiber filter membrane (used for 24 h without backwash) and (b) GO-modified PP fiber filter membrane combined with flocculation process (used for 24 h without backwash).

Table 2
Comparison of removal rate of SS in the initial operation and after backwashing

	Removal rate of SS (%)			
	PP fiber filter membrane	GO-PP fiber filter membrane	Flocculation + PP fiber filter membrane	Flocculation + GO-PP fiber filter membrane
Initial operation for 2 h	53.2	68.6	75.5	84.8
Operation for 2 h after backwash (used for 24 h)	53.0	68.1	75.7	84.3

and SS removal rates using flocculation + GO-PP fiber filter membrane unit was 84.8% and 84.6%, respectively. The membrane flux for GO-PP fiber filter membrane was reduced compared to PP fiber filter membrane due to the blockage between pores on the surface of PP fiber filter membrane and additional resistance caused by the GO layer.

The SEM-EDS characterization of samples indicated the membrane pollution of used filter membrane. This paper indicated the vast potential for the application of GO-PP fiber filter membrane in water-based paint wastewater treatment.

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